Nwamicha 10/5/1823

Page 1

=> fil reg
COST IN U.S. DOLLARS

SINCE FILE TOTAL ENTRY SESSION 0.21 0.21

FULL ESTIMATED COST

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STRUCTURE FILE UPDATES: 25 JUL 2005 HIGHEST RN 856925-80-9 DICTIONARY FILE UPDATES: 25 JUL 2005 HIGHEST RN 856925-80-9

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TSCA INFORMATION NOW CURRENT THROUGH JANUARY 18, 2005

Please note that search-term pricing does apply when conducting SmartSELECT searches.

Structure search iteration limits have been increased. See HELP SLIMITS for details.

Experimental and calculated property data are now available. For more information enter HELP PROP at an arrow prompt in the file or refer to the file summary sheet on the web at: http://www.cas.org/ONLINE/DBSS/registryss.html

=> e dihydroxydiphenylsulfone/cn 5

E1	1	DIHYDROXYDIPHENYLSILANE-N,N'-(4,4'-DIPHENYLMETHANE)BISMALEIM
		IDE COPOLYMER/CN
E2	1	DIHYDROXYDIPHENYLSILANE-TETRABUTOXYTITANIUM POLYMER/CN
E3	0>	DIHYDROXYDIPHENYLSULFONE/CN
E4	1	DIHYDROXYDIPHENYLTELLURIUM/CN
E5	1	DIHYDROXYDIPICOLINATE SYNTHASE (CHROMOBACTERIUM VIOLACEUM ST
•		RAIN ATCC 12472 GENE CV2825)/CN

=> e trihydroxytriphenylsulfone/cn 5

=>	е	trinyaroxytri	pnenyisulione/cn 5
E1		1	TRIHYDROXYSTEARIN/CN
E2		1	TRIHYDROXYTETRAFLUOROPROPANOL/CN
E3		0>	TRIHYDROXYTRIPHENYLSULFONE/CN
E4		1	TRIHYDROXYVINYLSILANE/CN
E5		1	TRII PROTEIN (YERSINIA PSEUDOTUBERCULOSIS STRAIN 1P32953 PLA
			SMID PYV GENE TRII)/CN

=> s dihydroxy(1)diphenylsulfone

358863 DIHYDROXY

403 DIPHENYLSULFONE

L1 47 DIHYDROXY (L) DIPHENYLSULFONE

=> s trihydroxy(l)triphenylsulfone

74472 TRIHYDROXY

0 TRIPHENYLSULFONE

L2 0 TRIHYDROXY(L)TRIPHENYLSULFONE

=> s trihydroxy(1)triphenylsulphone

74472 TRIHYDROXY

0 TRIPHENYLSULPHONE

L3 0 TRIHYDROXY (L) TRIPHENYLSULPHONE

=> s trihydroxy(1)?phenylsulfone

LEFT TRUNCATION IGNORED FOR '?PHENYLSULFONE' FOR FILE 'REGISTRY'

74472 TRIHYDROXY

434 PHENYLSULFONE

L4 0 TRIHYDROXY(L)?PHENYLSULFONE

Left truncation is not valid in the specified search field in the specified file. The term has been searched without left truncation. Examples: '?TERPEN?' would be searched as 'TERPEN?' and '?FLAVONOID' would be searched as 'FLAVONOID.'

If you are searching in a field that uses implied proximity, and you used a truncation symbol after a punctuation mark, the system may interpret the truncation symbol as being at the beginning of a term. Implied proximity is used in search fields indexed as single words, for example, the Basic Index.

=> fil medl,biosis,embase,caplus;s (l1 or dihydroxydiphenylsulfone or dihydroxydiphenylsulphone)

COST IN U.S. DOLLARS

SINCE FILE TOTAL
ENTRY SESSION
38.52 38.73

FULL ESTIMATED COST

FILE 'MEDLINE' ENTERED AT 12:15:10 ON 26 JUL 2005

FILE 'BIOSIS' ENTERED AT 12:15:10 ON 26 JUL 2005

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L5 3 FILE MEDLINE
L6 2 FILE BIOSIS
L7 1 FILE EMBASE
L8 1120 FILE CAPLUS

TOTAL FOR ALL FILES

L9 1126 (L1 OR DIHYDROXYDIPHENYLSULFONE OR DIHYDROXYDIPHENYLSULPHONE)

=> s trihydroxytriphenylsulfone or trihydroxy(1)triphenylsulfone or trihydroxy(1)triphenylsulphone

L10 0 FILE MEDLINE
L11 0 FILE BIOSIS

```
O FILE EMBASE
L12
L13
             0 FILE CAPLUS
TOTAL FOR ALL FILES
             O TRIHYDROXYTRIPHENYLSULFONE OR TRIHYDROXY(L) TRIPHENYLSULFONE OR
               TRIHYDROXY (L) TRIPHENYLSULPHONE
=> s ?triphenylsulfone? or ?triphenylsulphone?
             O FILE MEDLINE
L16
             0 FILE BIOSIS
L17
             O FILE EMBASE
L18
             1 FILE CAPLUS
TOTAL FOR ALL FILES
             1 ?TRIPHENYLSULFONE? OR ?TRIPHENYLSULPHONE?
=> s 19 and 119
             O FILE MEDLINE
L21
             0 FILE BIOSIS
L22
             0 FILE EMBASE
L23
             0 FILE CAPLUS
TOTAL FOR ALL FILES
             0 L9 AND L19
L24
=> s (dissolv? or suspend?) and 19
             O FILE MEDLINE
L26
             O FILE BIOSIS
L27
            O FILE EMBASE
L28
            46 FILE CAPLUS
TOTAL FOR ALL FILES
            46 (DISSOLV? OR SUSPEND?) AND L9
=> s alkali metal hydroxide and 129
             O FILE MEDLINE
L31
             O FILE BIOSIS
L32
             O FILE EMBASE
             0 FILE CAPLUS
L33
TOTAL FOR ALL FILES
             0 ALKALI METAL HYDROXIDE AND L29
=> s crude and 129
             O FILE MEDLINE
L35
L36
             0 FILE BIOSIS
L37
             O FILE EMBASE
L38
             4 FILE CAPLUS
TOTAL FOR ALL FILES
             4 CRUDE AND L29
=> d 1-4 ibib abs;s wakayama f?/au;s yanase n?/au;s kitahara t?/au;s nate n?/au
L39 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER:
                         2001:780846 CAPLUS
DOCUMENT NUMBER:
                         135:318322
TITLE:
                         Semi-continuous method for producing 4,4'-
                         dihydroxydiphenylsulfone from phenol and a
                         sulfonating agent in heated water
```

Prepared by: Mary Hale @2-2507 Rem Bldg 1D86

Page 3

INVENTOR(S):

Pabst, Gunther; Kast, Juergen

PATENT ASSIGNEE(S):

Basf A.-G., Germany
PCT Int. Appl., 16 pp.

SOURCE:

CODEN: PIXXD2

DOCUMENT TYPE:

Patent German

LANGUAGE:

Gern

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

				KIND DATE			APPLICATION NO.				DATE							
	WO	2001	0791	63					WO 2001-EP4081					20010410				
												BG,						
												ES,						
												KP,						
												MX,					-	
			-							•		TR,	•	•	•	•	•	•
												MD,	-			00,	00,	00,
		RW:		-		-		-	-	•		TZ,	•	•		BE	СН	CY
								•		-	•	LU,	•	•	•	•	•	•
												MR,			-	-	,	D.,
	DE	1001	-	-								000-		•	•		0000	414
		2001																
		1272																
		•										IT,						
								RO,					,	_,	,	,	,	,
	JР	2003										001-	5767	65		2	0010	410
		2003										002-					0021	
		6700									-					_	0021	00
PRIO		APP									DE 2	000-	1001	8580	;	A 2	0000	414
		- -			-							1001-1						

OTHER SOURCE(S): CASREACT 135:318322

AB A semi-continuous method for producing 4,4'-

dihydroxydiphenylsulfone comprises: (a) reacting phenol with a sulfonating agent (e.g., concentrate sulfuric acid); (b) suspending the resulting crude product in ≥40° water which is

free from inert organic solvents and can contain residual amts. of unreacted phenol, and filtering off the product; and (c) returning the resulting waste streams containing the educt and/or product to the production process.

Step

(b) is carried out using the **crude** product and water in a weight ratio of 85:15 to 55:45.

REFERENCE COUNT:

THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L39 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER:

1995:356764 CAPLUS

DOCUMENT NUMBER:

122:119046

TITLE:

A heat-sensitive recording material.

INVENTOR(S):

Kobayashi, Norio; Takahashi, Toshiaki; Makino,

Masahiro; Hosoda, Masaaki

PATENT ASSIGNEE(S):

Nicca Chemical Co., Ltd., Japan

SOURCE:

Eur. Pat. Appl., 11 pp.
CODEN: EPXXDW

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.

KIND DATE

APPLICATION NO.

DATE

19940928 EP 1994-104540 19940323 ----------EP 616897 A2 19940928 EP 616897 A3 19941214 EP 616897 B1 19990616 R: CH, DE, FR, GB, IT, LI JP 06270550 A2 19940927 JP 1993-89426 19930324 US 5378674 A 19950103 US 1994-216379 19940323 RITY APPLN. INFO.: JP 1993-89426 A 19930324 PRIORITY APPLN. INFO.: A heat-sensitive recording material comprises a heat-sensitive color forming layer which is formed on a supporter and contains a colorless or light color leuco dyestuff as a color forming substance, a developer which develops color of the leuco dyestuff by reaction with it when heated and a sensitizer. The developer is 2,4'-dihydroxydiphenylsulfone having purity of 97% or more and prepared by washing and drying crystal which is obtained by dissolving crude 2,4'dihydroxydiphenylsulfone in an alc. having 1 to 4 C atoms or in a mixture of an alc. having 1 to 4 C atoms and H2O by heating and then cooling the solution or partially removing the solvent from the solution by distillation The heat-sensitive recording material has excellent properties, such as reduced fog and excellent image preservation (weatherability). L39 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2005 ACS on STN ACCESSION NUMBER: 1990:7167 CAPLUS DOCUMENT NUMBER: 112:7167 TITLE: Process for the purification and isolation of mixtures of 4,4'- and 2,4'-dihydroxydiphenylsulphone INVENTOR(S): Arient, Josef Czech. PATENT ASSIGNEE(S): SOURCE: Czech., 3 pp. CODEN: CZXXA9 DOCUMENT TYPE: Patent LANGUAGE: Czech FAMILY ACC. NUM. COUNT: 1 PATENT INFORMATION: KIND DATE APPLICATION NO. PATENT NO. --------------______ B1 19880415 CS 1986-6143 19860822 CS 257071 PRIORITY APPLN. INFO :: CS 1986-6143 19860822 PhOH is sulfonated at 180-190°, the **crude** product is dissolved in hot aqueous NaOH, and the solution is boiled with C to remove resinous and colored contaminants. The hot filtrate is decolorized with a 2-5% aqueous Na2S2O5 solution and product (70%) containing the title compds. is separated with HCl from a cooled solution L39 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2005 ACS on STN ACCESSION NUMBER: 1946:32301 CAPLUS DOCUMENT NUMBER: 40:32301 ORIGINAL REFERENCE NO.: 40:6281c-e TITLE: U.S. Government reports disclose German process developments Curtis, Francis J.; Fogler, F. AUTHOR (S): CORPORATE SOURCE: I.G. Farbenindustrie A.-G. Elherfeld and Leverkusen SOURCE: Shoe and Leather Reporter (1946), 241(No. 11), 29-30 CODEN: SLREAY; ISSN: 0096-9257 DOCUMENT TYPE: Journal LANGUAGE: Unavailable Tanigan Extra A is a mixture of 4,4-dihydroxydiphenylsulfone (I)

and dihydroxydiphenylsulfone formaldehyde resin (II) in sulfite liquor (III). To prepare III treat raw CaHSO3 waste liquor at 50° with 50% NaOH to pH 8.6, then at 55° with NaOH until no further precipitate of lime forms. Mix 0.5 hr., filter, let settle 12 hrs. until Ca content is less than 0.1%, then concentrate to 53% solids. To prepare I run 1600

I. of H2SO4.H2O into 9400 l. of crude phenol at 65° in 3 hrs.; heat under reduced pressure to 150°, distilling off 5000 l. of excess phenol and H2O in 30 hrs. Neutralize and dissolve in 520 l. of 50% NaOH and 2600 l. of H2O under pressure. The resin is prepared by stirring together 11,600 l. of III and 800 l. of I at 110°, adjusting to an alkali number of 4.0, then, at 65°, adding the necessary HCHO (approx. 60 l. per 100 kg. of sulfone) in 20 min. and heating to 105° until condensation is complete. Yield: 4.4 to 4.8 times the amount of phenol. Brief descriptions of preparation of Tanigans Extra

B and Extra E are given.

```
3 FILE MEDLINE
L40
L41
             1 FILE BIOSIS
T.42
             2 FILE EMBASE
T.43
             1 FILE CAPLUS
TOTAL FOR ALL FILES
L44
            7 WAKAYAMA F?/AU
         32 FILE MEDLINE
1.45
            26 FILE BIOSIS
1.46
L47
            29 FILE EMBASE
L48
           188 FILE CAPLUS
TOTAL FOR ALL FILES
1.49
           275 YANASE N?/AU
L50
           369 FILE MEDLINE
L51
           458 FILE BIOSIS
L52
           326 FILE EMBASE
           994 FILE CAPLUS
TOTAL FOR ALL FILES
L54
          2147 KITAHARA T?/AU
L55
             O FILE MEDLINE
L56
             5 FILE BIOSIS
L57
             O FILE EMBASE
L58
            21 FILE CAPLUS
TOTAL FOR ALL FILES
            26 NATE N?/AU
=> s oi f?/au
L60
            7 FILE MEDLINE
L61
            10 FILE BIOSIS
L62
             2 FILE EMBASE
L63
            28 FILE CAPLUS
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TOTAL FOR ALL FILES
L64
              47 OI F?/AU
=> s 164 and 159 and 154 and 149
               O FILE MEDLINE
L65
L66
               O FILE BIOSIS
L67
               O FILE EMBASE
L68
               2 FILE CAPLUS
TOTAL FOR ALL FILES
               2 L64 AND L59 AND L54 AND L49
=> d 1-2 ibib abs
L69 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER:
                             2003:875243 CAPLUS
DOCUMENT NUMBER:
                             139:350539
TITLE:
                             Process for producing dihydroxydiphenyl sulfone by
                             crystallization
INVENTOR (S):
                             Oi, Fumio; Yanase, Norio;
                             Kitahara, Takayuki; Nate, Nobuyuki
PATENT ASSIGNEE(S):
                             Konishi Chemical Ind. Co., Ltd., Japan
SOURCE:
                             PCT Int. Appl., 12 pp.
                             CODEN: PIXXD2
DOCUMENT TYPE:
                             Patent
LANGUAGE:
                             Japanese
FAMILY ACC. NUM. COUNT:
PATENT INFORMATION:
                                                   APPLICATION NO.
      PATENT NO.
                             KIND
                                     DATE
                                                                              DATE
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                                      -----
                                                   -----
                                     20031106
                                                  WO 2003-JP5228
      WO 2003091206
                             A1
                                                                              20030424
               AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
               CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
               GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PH,
          PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
      EP 1498412
                                      20050119
                                                 EP 2003-725653
                              Α1
                                                                              20030424
               AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
               IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK
                                                    JP 2002-123646
PRIORITY APPLN. INFO.:
                                                                          A 20020425
                                                   WO 2003-JP5228
                                                                           W 20030424
      Disclosed is a process for producing dihydroxydiphenyl sulfone in which
      only trihydroxytriphenyl disulfone and coloring impurities are effectively
      removed without changing the proportion of dihydroxydiphenyl sulfone
                 The process for producing dihydroxydiphenyl sulfone is
      characterized by dissolving or suspending crude dihydroxydiphenyl sulfone
      containing trihydroxytriphenyl disulfone in an aqueous solvent, regulating the
pН
      of the solution or suspension to 5 to 7, optionally cooling it, and separating
out
      the dihydroxydiphenyl sulfone crystals precipitated This process is superior
in
      handlability, safety, sanitation, and cost effectiveness since it uses
```

water instead of organic solvent. Thus, a mixture of 4,4'-dihydroxydiphenyl sulfone 75, 2,4'-dihydroxydiphenyl sulfone 20, and trihydroxytriphenyl disulfone 5 weight% (100 g containing 0.39 mol 4,4'- and 2,4'-dihydroxydiphenyl sulfone and trihydroxytriphenyl disulfone, APHA 1,000 in acetone solution) was treated with 300 g H2O and 8 g NaOH (0.2 mol, 0.5-times mole vs. the sulfones), dissolved under heating at 90°, adjusted to pH 6.5 by adding 50% aqueous H2SO4, and cooled to 35°, followed by filtration of the precipitated crystals, washing with water, and drying to give 92 g dry crystals containing 4,4'-dihydroxydiphenyl sulfone 78.9, 2,4'dihydroxydiphenyl sulfone 21.0, and trihydroxytriphenyl disulfone 0.1 weight% (APHA 400 in acetone solution).

REFERENCE COUNT:

THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L69 ANSWER 2 OF 2 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 2003:841197 CAPLUS

DOCUMENT NUMBER:

139:343510

TITLE:

Process for manufacturing mixture of

dihydroxydiphenylsulfone isomers Ogata, Eiji; Oi, Fumio; Yanase,

Norio; Nate, Nobuyuki; Kitahara,

Takayuki

PATENT ASSIGNEE(S):

Konishi Kagaku Kogyo Co., Ltd., Japan

SOURCE:

Jpn. Kokai Tokkyo Koho, 6 pp.

CODEN: JKXXAF

DOCUMENT TYPE:

LANGUAGE:

INVENTOR(S):

Patent Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE

JP 2003306477	A2	20031028	JP 2002-352839	20021204
י הׄדות הוזממג עידומהדמ			TD 2002-38473 A	20020215

The title process comprises heating a crude mixture of 2,4'dihydroxydiphenylsulfone (I), 4,4'-dihydroxydiphenylsulfone (II), water, and an alkali (0.55 equiv relative to the total amount of I and II), cooling the mixture, separating the crystals of II, and adding an acid to the separated liquid

The mixture obtained by the title process contains 25 to 50 weight% I. I and II are developers for thermal recording material.

=> fil reg

COST IN U.S. DOLLARS SINCE FILE TOTAL SESSION ENTRY FULL ESTIMATED COST 64.51 103.24 DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) SINCE FILE TOTAL ENTRY SESSION CA SUBSCRIBER PRICE -4.38 -4.38

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Page 9

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STRUCTURE FILE UPDATES: 25 JUL 2005 HIGHEST RN 856925-80-9 DICTIONARY FILE UPDATES: 25 JUL 2005 HIGHEST RN 856925-80-9
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TSCA INFORMATION NOW CURRENT THROUGH JANUARY 18, 2005

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Experimental and calculated property data are now available. For more information enter HELP PROP at an arrow prompt in the file or refer to the file summary sheet on the web at: http://www.cas.org/ONLINE/DBSS/registryss.html

```
=> e "2,4'-dds"/cn 5
                   2,4'-CYCLOHEXYLIDENEDIPHENOL/CN
                   2,4'-DDE/CN
E2
              --> 2,4'-DDS/CN
E3
                   2,4'-DDT/CN
E4
             1
                   2,4'-DI(SEC-BUTYLAMINOPHENYL) ETHER/CN
E5
=> e "4,4'-dds"/cn 5
                   4,4'-DDD/CN
E1
             1
                   4,4'-DDE/CN
E2
              --> 4,4'-DDS/CN
E3
E4
             1
                   4,4'-DDT/CN
                   4,4'-DECAMETHYLENEBIS(1,1-DIETHYLPIPERAZINIUM IODIDE)/CN
E5
=> e "2,4'-dihydroxydiphenylsulfone"/cn
                   2,4'-DIHYDROXYDIPHENYLMETHANE-4,4'-DIHYDROXYDIPHENYLMETHANE-
E1
                   PHENOL-FORMALDEHYDE POLYMER/CN
E2
                   2,4'-DIHYDROXYDIPHENYLMETHANE-FORMALDEHYDE COPOLYMER/CN
             0 --> 2,4'-DIHYDROXYDIPHENYLSULFONE/CN
E3
                   2,4'-DIISOCYANATO-1,1'-BICYCLOHEXYL/CN
E4
             1
                   2,4'-DIISOCYANATO-1,2-DIPHENYLETHANE/CN
E5
             1
                   2,4'-DIISOCYANATO-3'-(ETHYLMERCAPTO)DIPHENYL SULFIDE/CN
E6
             1
E7
             1
                   2,4'-DIISOCYANATO-3'-CHLORODIPHENYL SULFIDE/CN
                   2,4'-DIISOCYANATO-3'-CHLORODIPHENYL SULFONE/CN
E8
             1
                   2,4'-DIISOCYANATO-3'-ETHYLDIPHENYL SULFIDE/CN
             1
E9
             1
                   2,4'-DIISOCYANATO-5-METHOXYDIPHENYL SULFIDE/CN
E10
                   2,4'-DIISOCYANATODIPHENYL ETHER/CN
E11
             1
             1
                   2,4'-DIISOCYANATODIPHENYL SULFIDE/CN
E12
=> e "2,4'-dihydroxydiphenyl sulfone"/cn
                   2,4'-DIHYDROXYCHALCONE/CN
E1
             1
                   2,4'-DIHYDROXYDIBENZOYLMETHANE/CN
E2
             1 --> 2,4'-DIHYDROXYDIPHENYL SULFONE/CN
E3
```

Page 10							
E4	2,4'-DIHYDROXYDIPHENYL SULFONE-4,4'-DIHYDROXYDIPHENYL SULFON E-FORMALDEHYDE COPOLYMER/CN						
E 5	1 2,4'-DIHYDROXYDIPHENYL SULFONE-4,4'-DIHYDROXYDIPHENYL SULFON						
E6	E-FORMALDEHYDE-P-PHENOLSULFONIC ACID COPOLYMER/CN 2,4'-DIHYDROXYDIPHENYL SULFONE-4,4'-DIHYDROXYDIPHENYL SULFON						
E7	E-PHENYLDICHLOROPHOSPHINE OXIDE POLYMER/CN 1 2,4'-DIHYDROXYDIPHENYLAMINE/CN						
E8	1 2,4'-DIHYDROXYDIPHENYLDIMETHYLMETHANE/CN						
E9	1 2,4'-DIHYDROXYDIPHENYLMETHANE/CN						
E10	1 · 2,4'-DIHYDROXYDIPHENYLMETHANE-4,4'-DIHYDROXYDIPHENYLMETHANE- PHENOL-FORMALDEHYDE POLYMER/CN						
E11	1 2,4'-DIHYDROXYDIPHENYLMETHANE-FORMALDEHYDE COPOLYMER/CN						
E12	2,4'-DIISOCYANATO-1,1'-BICYCLOHEXYL/CN						
=> s e3-e6							
	1 "2,4'-DIHYDROXYDIPHENYL SULFONE"/CN 1 "2,4'-DIHYDROXYDIPHENYL SULFONE-4,4'-DIHYDROXYDIPHENYL SULFONE-F						
	ORMALDEHYDE COPOLYMER"/CN						
	1 "2,4'-DIHYDROXYDIPHENYL SULFONE-4,4'-DIHYDROXYDIPHENYL SULFONE-F						
	ORMALDEHYDE-P-PHENOLSULFONIC ACID COPOLYMER"/CN 1 "2,4'-DIHYDROXYDIPHENYL SULFONE-4,4'-DIHYDROXYDIPHENYL SULFONE-P						
	HENYLDICHLOROPHOSPHINE OXIDE POLYMER"/CN						
L70	4 ("2,4'-DIHYDROXYDIPHENYL SULFONE"/CN OR "2,4'-DIHYDROXYDIPHENYL						
	SULFONE-4,4'-DIHYDROXYDIPHENYL SULFONE-FORMALDEHYDE COPOLYMER"/C N OR "2,4'-DIHYDROXYDIPHENYL SULFONE-4,4'-DIHYDROXYDIPHENYL						
	SULFONE-FORMALDEHYDE-P-PHENOLSULFONIC ACID COPOLYMER"/CN OR						
	"2,4'-DIHYDROXYDIPHENYL SULFONE-4,4'-DIHYDROXYDIPHENYL SULFONE-P						
	HENYLDICHLOROPHOSPHINE OXIDE POLYMER"/CN)						
=> e "4,4'-d	ihydroxydiphenyl sulfone"/cn 5						
E1	1 4,4'-DIHYDROXYDIPHENYL SULFIDE-ISOPHTHALOYL DICHLORIDE-TEREP						
E2	HTHALOYL DICHLORIDE COPOLYMER, SRU/CN 1 4,4'-DIHYDROXYDIPHENYL SULFIDE-TEREPHTHALOYL CHLORIDE COPOLY						
	MER, SRU/CN						
E3	1> 4,4'-DIHYDROXYDIPHENYL SULFONE/CN 1 4,4'-DIHYDROXYDIPHENYL SULFONE BIS(DOCOSANOATE)/CN						
E4 E5	1 4,4'-DIHIDROXIDIPHENIL SULFONE BIS (DOCOSANOATE)/CN 1 4,4'-DIHYDROXYDIPHENYL SULFONE BISFLUOROSULFATE/CN						
=> s e3 L71	1 "4,4'-DIHYDROXYDIPHENYL SULFONE"/CN						
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<pre>=> fil medl, COST IN U.S.</pre>	biosis, embase, caplus; s 170 or 171						
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L72
             O FILE MEDLINE
            32 FILE BIOSIS
L73
L74
             O FILE EMBASE
1.75
          1750 FILE CAPLUS
TOTAL FOR ALL FILES
L76
          1782 L70 OR L71
=> s "4,4'-dihydroxydiphenyl sulfone"
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L78
             3 FILE EMBASE
· L79
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L80
TOTAL FOR ALL FILES
           755 "4,4'-DIHYDROXYDIPHENYL SULFONE"
L81
=> s "2,4'-dihydroxydiphenyl sulfone" ?
ADDITIONAL CHARACTERS REQUIRED AFTER '?' FOR LEFT TRUNCATION
Additional characters must follow the left truncation symbol in your
search term. If your search term contains a punctuation mark before
the truncation symbol and you are searching in a field that uses
implied proximity, the system may interpret the truncation symbol as
being at the beginning of a term. Implied proximity is used in search
fields indexed as single words, for example, the Basic Index. To see
which fields in the current file have left truncation, enter "HELP
SFIELDS" at an arrow prompt (=>).
=> s "2,4'-dihydroxydiphenyl sulfone"
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L82
L83
             0 FILE BIOSIS
L84
             O FILE EMBASE
L85
           170 FILE CAPLUS
TOTAL FOR ALL FILES
L86
           170 "2,4'-DIHYDROXYDIPHENYL SULFONE"
=> s (176 or 181 or 186) and (make or making or process?\ or produc?)
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=> s (176 or 181 or 186) and (make or making or process? or produc?)
             O FILE MEDLINE
L88
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             8 FILE BIOSIS
L90
             1 FILE EMBASE
L91
           737 FILE CAPLUS
TOTAL FOR ALL FILES
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=> s tri hydroxy triphenyl sulfone or trihydroxy triphenyl sulfone or
trihydroxytriphenyl sulfone
L93
             O FILE MEDLINE
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Page 12
L95
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             0 FILE CAPLUS
L96
TOTAL FOR ALL FILES
             0 TRI HYDROXY TRIPHENYL SULFONE OR TRIHYDROXY TRIPHENYL SULFONE
1.97
               OR TRIHYDROXYTRIPHENYL SULFONE
=> s triphenylsulfone or triphenyl sulfone or trihydroxy(1) (sulfone or sulphone)
             O FILE MEDLINE
L99
             0 FILE BIOSIS
L100
             1 FILE EMBASE
L101
             6 FILE CAPLUS
TOTAL FOR ALL FILES
             7 TRIPHENYLSULFONE OR TRIPHENYL SULFONE OR TRIHYDROXY(L) (SULFONE
L102
               OR SULPHONE)
=> s 1102 and 192
             O FILE MEDLINE
L103
L104
             O FILE BIOSIS
L105
             O FILE EMBASE
L106
             O FILE CAPLUS
TOTAL FOR ALL FILES
L107
            0 L102 AND L92
=> s 192 and crystal? and crude
             O FILE MEDLINE
             0 FILE BIOSIS
L109
             O FILE EMBASE
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             8 FILE CAPLUS
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L112
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L113
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L114
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L115
             5 FILE CAPLUS
L116
TOTAL FOR ALL FILES
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L117
=> d 1-5 ibib abs
L117 ANSWER 1 OF 5 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER:
                     2004:758798 CAPLUS
DOCUMENT NUMBER:
                         141:277350
TITLE:
                         Process for preparing mixture of
                         dihydroxydiphenylsulfone isomers
INVENTOR(S):
                         Oi, Satsuo; Yanase, Norio; Nate, Nobuyuki; Nagaoka,
                         Etsuzo
PATENT ASSIGNEE(S):
                         Konishi Kagaku Kogyo Co., Ltd., Japan
SOURCE:
                         Jpn. Kokai Tokkyo Koho, 8 pp.
                         CODEN: JKXXAF
DOCUMENT TYPE:
                         Patent
                         Japanese
LANGUAGE:
FAMILY ACC. NUM. COUNT:
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Prepared by: Mary Hale @2-2507 Rem Bldg 1D86

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE

JP 2004256422 A2 20040916 JP 2003-47540 20030225
PRIORITY APPLN. INFO.: JP 2003-47540 20030225

AB In the manufacture of a mixture of 2,4'-dihydroxydiphenylsulfone (I) and 4,4'-dihydroxydiphenylsulfone (II) containing 10 weight% to 90 weight% I, said mixture

of crude dihydroxydiphenylsulfone containing phenolsulfonic acid Ph ester (III) as impurity and a mixture of water and lower alc. (IV) containing ≥ 2 weight% IV are mixed and heated and then cooled, and the precipitating crystals are separated at pH 4 to 8. Thus, a mixture of crude II and I (II/I ratio = 67/33) containing 2.9 weight% III, water, methanol, and sodium hydroxide was stirred and heated until a solution was obtained at 69°C; said solution was cooled to 30°C to give crystals of I and II containing only 0.8 weight% III; the pH of said solution before the collection of the crystals was 6.8. A high quality heat-sensitive recording paper was produced using the title mixture

L117 ANSWER 2 OF 5 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 2004:756320 CAPLUS

DOCUMENT NUMBER: 141:277349

TITLE: Method for manufacturing a mixture of

dihydroxydiphenylsulfone isomers

INVENTOR(S): Oi, Satsuo; Yanase, Norio; Nate, Nobuyuki; Nagaoka,

Etsuzo

PATENT ASSIGNEE(S): Konishi Kagaku Kogyo Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. -----_____ ---------JP 2004256421 A2 20040916 JP 2003-47538 20030225 PRIORITY APPLN. INFO.: JP 2003-47538 20030225 In the manufacture of a mixture of 2,4'-dihydroxydiphenylsulfone (I) and

4,4'-dihydroxydiphenylsulfone (II) containing 10 weight% to 90 weight% I, said mixture

of crude dihydroxydiphenylsulfone containing phenolsulfonic acid Phester (III) as impurity, an alkaline substance (e.g., sodium hydroxide) at 0.02 to 0.4 equiv (relative to dihydroxydiphenylsulfone), and an aqueous solvent (e.g., water) are heated and mixed and then cooled, and the precipitating

crystals are separated The title method is industrially advantageous.
Thus, a mixture of crude II and I (II/I ratio = 67/33) containing 2.9
weight% III, water, and sodium hydroxide was stirred and heated until a
solution

was obtained at 92°C; said solution was cooled to 60°C and kept at 60°C for 1 h to give **crystals** of I and II containing only 0.4 weight% III. A high quality heat-sensitive recording paper was **produced** using the title mixture

L117 ANSWER 3 OF 5 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 2003:678778 CAPLUS

DOCUMENT NUMBER: 139:230468

TITLE: Process for preparation of

dihydroxydiphenylsulfone isomeric mixtures Oi, Fumio; Yanase, Norio; Nate, Nobuyuki INVENTOR (S): Konishi Chemical Ind. Co., Ltd., Japan PATENT ASSIGNEE(S): PCT Int. Appl., 20 pp. SOURCE: CODEN: PIXXD2 DOCUMENT TYPE: Patent LANGUAGE: Japanese FAMILY ACC. NUM. COUNT: PATENT INFORMATION: APPLICATION NO. DATE PATENT NO. KIND DATE -----_____ ----20030220 20030828 WO 2003070695 A1 WO 2003-JP1836 W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG JP 2003313160 20031106 JP 2002-319967 A2 20021101 JP 2002-46629 PRIORITY APPLN. INFO.: A 20020222 JP 2002-319967 A 20021101 OTHER SOURCE(S): CASREACT 139:230468 This invention pertains to a method for producing high-quality dihydroxydiphenylsulfone isomeric mixts. which cause color development (color formation) in non-image areas when used in thermal recording paper as the developer. Specifically, a process for the prodn . of dihydroxydiphenylsulfone isomeric mixts., characterized by subjecting a solution or suspension of a crude isomeric mixture comprising 2,4'-dihydroxydiphenylsulfone and 4,4'-dihydroxydiphenylsulfone in an organic solvent to cooling and filtration successively; a process for the production of dihydroxydiphenylsulfone isomeric mixts., characterized by mixing a solution or suspension of a crude isomeric mixture comprising 2,4'-dihydroxydiphenylsulfone and 4,4'-dihydroxydiphenylsulfone in an organic solvent with an aqueous basic solution to extract the isomeric mixture into the aqueous basic solution, removing the resulting organic solvent layer by liquid-liquid separation, adding an acid to the resulting aqueous basic solution to precipitate crystals, and recovering the crystals by filtration. For example, phenol was treated with concentrate H2SO4 in 1,2-dichlorobenzene to give a mixture of 2,4'dihydroxydiphenylsulfone and 4,4'-dihydroxydiphenylsulfone (35/65). REFERENCE COUNT: THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT L117 ANSWER 4 OF 5 CAPLUS COPYRIGHT 2005 ACS on STN ACCESSION NUMBER: 1988:610700 CAPLUS DOCUMENT NUMBER: 109:210700 TITLE: Synthesis of bisphenol S INVENTOR(S): Cui, Xianghao; Wang, Yubin; et al.

Jilin University, Peop. Rep. China

CODEN: CNXXEV

Patent

Chinese

Faming Zhuanli Shenqing Gongkai Shuomingshu, 4 pp.

Prepared by: Mary Hale @2-2507 Rem Bldg 1D86

PATENT ASSIGNEE(S):

DOCUMENT TYPE:

SOURCE:

LANGUAGE:

FAMILY ACC. NUM. COUNT: 1 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CN 87100796	A	19870902	CN 1987-100796	19870218
CN 87100796	В	19880907		
PRIORITY APPLN. INFO.:			CN 1987-100796	19870218
GT				

$$_{\text{HO}}$$
 $_{\text{SO}_2}$ $_{\text{OH}}$ $_{\text{I}}$

AB Bisphenol S (I), a widely useful industrial chemical, is prepared in an economical process without environmental pollution. A mixture of PhOH 198, com. H2SO4 100, and recovered mother liquor from a previous run 287 g was heated 3 h at 190°, cooled to 160°, 10-30% EtOH added at 90°, the solution cooled to 30-50° to precipitate 240 g I and 400 g mother liquor. The crude I of 97% purity was purified through activated C to give I of 99.8% purity.

L117 ANSWER 5 OF 5 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1960:97352 CAPLUS

DOCUMENT NUMBER: 54:97352

ORIGINAL REFERENCE NO.: 54:18409d-i,18410a-i,18411a-i,18412a-g

TITLE: Action of thiols and sulfinic acids on quinol

acetates. II

AUTHOR(S): Wessely, F.; Swoboda, J.; Schmidt, G.

CORPORATE SOURCE: Univ. Vienna

SOURCE: Monatshefte fuer Chemie (1960), 91, 57-78

CODEN: MOCMB7; ISSN: 0026-9247

DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
CL For diagram(s) see printed CA Issue

GI For diagram(s), see printed CA Issue.

cf. CA 51, 12848a. In continuation of prior work (loc. cit.), the action of MeSH, NaSH, H2S, PhSH, MeSO2H, and PhSO2H with various o-quinol acetates (I) in the presence of various bases in different solvents was investigated. The above thiols gave m- or o-substituted phenols in addition to p-substituted phenols resulting from addition, cleavage of AcOH, and rearrangement reactions. MeSO2H and PhSO2H gave chiefly m-substituted phenols. The following I, CR''': CR''. CR': CH. CR (OAc). CO were used (R, R', R'', R''' given): Me, H, H, H (II); Me, Me, H, H (III); Me, H, Me, H (IV); Me, Me, H, Me (V); Me, H, H, Me (VI); Et, H, H, Et (VII); Et, H, Ph, Et (VIII). The following results were obtained with the I and MeSH [I used, solvent, base, % o-substituted phenol, % m-substituted phenol, % p-substituted phenol, % reduction product (this was identical with that phenol which on oxidation with Pb(OAc)4 gave the I used), sum of identified reaction products (%), reaction time given]: II, CH2Cl2, Et3N, 15 2,6-Me(MeS)C6H3OH (IX), 12 2,5-Me(MeS)C6H3OH (X), 69 2,4-Me(MeS)C6H3OH (XI), trace, 96, several days; II, CH2Cl2, Et3N, 13 IX, 11 X, 67 XI, trace, 91, 1 day; II, CH2Cl2, Ph3P, 11 IX, 3 X, 40 XI, trace, 54, several days; II, MeOH, Et3N, 10 IX, 34 X, 49 XI, trace, 93, several days; II, MeOH, MeONa, 7 IX, 47 X, 34 XI, trace, 88, 1 hr.; III, CH2Cl2, Et3N, 28 2,4,6-Me2(MeS)C6H2OH (XII), 5 2,4,5-Me2(MeS)C6H2OH (XIII), -, 57, 90, several days; III, MeOH, MeONa, 13 XII, 44 XIII, -, 38, 95, 1 hr.; V, CH2Cl2, Et3N, -, 14 2,4,6,3-Me3(MeS)C6HOH (XIV), -, 80, 94, several days;

V, MeOH, MeONa, -, 42 XIV, -, 54, 96, 1 hr.; VI, CH2Cl2, Et3N, -, 6 2,6,3-Me2(MeS)C6H2OH (XV), 64 2,6,4-Me2(MeS)C6H2OH (XVI), 18, 88, several days; VI, MeOH, MeONa, -, 28 XV, 38 XVI, trace, 66, 1 hr. Treatment of the I with NaSH gave the following results [I used, % o-, % m-, and % p-substituted phenol, resp., % reduction product, sum of identified reaction products (%) given]: II, 5 2,6-Me(HS)C6H3OH (XVII), -20 2,4-Me(HS)C6H3OH (XVIII), 23, 55 {in addition 7% [3,4-Me(HO)C6H3]2S was isolated as the sulfone (XIX) (see below)]; III, -, 10 2,4,6-Me2(HS)C6H2OH (XX), -, 65, 75; V, -, trace 2,4,6,3-Me3(HS)C6HOH, -, 79, 79; VI, 20 2,6,4-Me2(HS)C6H2OH (XXI), -, -, 38, 77 {in addition 19% [3,5,4-Me2(HO)C6H2]2SO2 (XXII) was isolated as sulfone (see below) }. of the I with MeSO2H gave the following results (I used and % product obtained given): II, 91 2,5-Me(MeSO2)C6H3OH (XXIII); III, 94 2,4,5-Me2(MeSO2)C6H2OH (XXIV); IV, 16 2,5,4-Me2(MeSO2)C6H2OH (XXV); V, 88 2,4,6,3-Me3(MeSO2)C6HOH (XXVI); VI, 90 2,6,3-Me2(MeSO2)C6H2OH (XXVII). Treatment of the I with PhSH gave the following results (all products were isolated as sulfones) (I used, solvent, base, % o-, % m-, and % p-substituted phenol, resp., % reduction product, remarks given): II, MeOH, MeONa, -, 2,5-Me(PhSO2)C6H3OH (XXVIII), 0.8 2,4-Me(PhSO2)C6H3OH (XXIX), -, in addition 1% mixture probably of XXVIII and XXIX was obtained; III, MeOH, MeONa, 2,4,6-Me2(PhSO2)C6H2OH (XXX), 12 2,4,5-Me2(PhSO2)C6H2OH (XXXI), -, 30, in addition 5% mixture of XXX and XXXI was obtained; V, MeOH, MeONa, -, 6 2,4,6,3-Me3(PhSO2)C6HOH, -, 25, -; VI, MeOH, MeONa, -, 2,6,3-Me2(PhSO2)C6H2OH (XXXII), 2,6,4-Me2(PhSO2)C6H2OH (XXXIII), 2,3% mixture of XXXII and XXXIII was obtained; VI, CH2Cl2, Et3N, -, -, 62 XXXIII, 19, -; VII, CHCl3, Et3N, -, -, 51 2,6,4-Et2(PhSO2)C6H2OH, -. The following results were obtained with the I and PhSO2H (I used and % product obtained given): VI, 68 XXXII; VII, 79 2,6,3-Et2(PhSO2)C6H2OH (XXXIV); VIII, 30 2,6,3,4-Et2Ph(PhSO2)C6HOH (XXXV). In the following exptl. work, methylation was accomplished in the usual way with excess Me2SO4 and aqueous NaOH at room temperature Oxidns. were effected

by dissolving the corresponding thioether in a little AcOH, adding 1.5 times the calculated amount of 30% H2O2 (if the thioether precipitated, it was redissolved by dropwise addition of AcOH), allowing the mixture to stand overnight at room temperature, warming 30 min. on a H2O bath, precipitating the product with H2O, extracting oily product with CH2Cl2,

washing the extract with saturated aqueous NaHCO3, drying, and evaporating; the residues

containing sulfones with free OH groups were crystallized by rubbing with Et2O; sulfones without free OH groups were distilled in vacuo; if crystalline product precipitated from the oxidation mixture, the mixture was kept several hrs. in a refrigerator, the precipitate filtered off, and washed

peroxide-free with dilute AcOH. In the reactions of the I with MeSH, NaSH, and H2S, mixts. of isomers were obtained in many cases, which were not quant. separable by the usual methods. In these cases, the yields given above were determined from other data. The compds. were identified by mixed m.ps., vapor phase chromatography, and m.p. diagrams with pure compds. or related derivs. The I dissolved in just the required amount of absolute MeOH

at room temperature, the solution added dropwise with stirring during 1 hr. to MeSH in

MeOH-MeONa (content 0.5 g. MeSH, 0.53 g. Na in 10 ml. MeOH), the mixture allowed to stand 1 hr., poured into 3 vols. H2O, acidified with HCl, extracted with Et2O, the extract washed with H2O, dried, evaporated, and the residue distilled

gave a distillate whose composition was determined by gas chromatography. The separation

of the reduction products and the o-substituted phenols from m- and

p-substituted phenols was accomplished by fractional distillation An analogous procedure was used for the isomeric mixture obtained from the I and H2S and NaSH. The I dissolved in just the required amount of solvent at room temperature,

the solution treated with MeSH (2-4 moles/mole I) and Et3N (0.05-0.1 ml./g. I), the mixture allowed to stand several days in a bomb tube, the solvent and excess MeSH evaporated, the residue taken up in Et2O, the Et2O solution washed with dilute HCl and saturated aqueous NaHCO3, and worked up as above gave the

products. The I with Ph3P (0.4 g./g. I) treated as in the Et3N
expts., the solvent and excess MeSH evaporated, the residue taken up in dilute
aqueous NaOH, the insol. Ph3P extracted with CH2Cl2, and the aqueous alkaline
phase worked

up as in the Et3N experiment gave the products. The isolation and properties of only the newly prepared compds. here and below were as follows. A mixture of 15% X and 85% XI oxidized (H2O2) and the mixture recrystd. from dilute AcOH gave 2,4-Me(MeSO2)C6H3OH, m. 126-7°, methylated to the Me ether. Crude XIII, obtained by dlstn., recrystd. from petr. ether gave XIII, m. 51-2°; Me ether (XXXVI) m. 50-1° (petr. ether). Oxidation of XIII gave XXIV, m. 139-40° (dilute AcOH); Me ether (by oxidation of XXXVI) m. 122-4° (dilute AcOH). From the reaction of IV with MeSH with Et3N was isolated 42% unidentified compound, C12H20O3S, m. 127-8° (MeOH), v (CCl4) 1755, 1237, and 1089 cm.-1 Crude XIV recrystd. from petr. ether gave XIV, m. 58-9°; Me ether bl0 130-50°. Oxidation of XIV gave XXVI, m. 154-5°. The I dissolved in a little absolute MeOH, the solution added dropwise with stirring during 1 hr. to 20% NaSH-absolute MeOH, the mixture allowed to stand 1 hr., poured into 3 vols. H2O, acidified, exhaustively extracted with Et20, the extract evaporated, and the product distilled to 150°/0.3 mm. gave a residue (larger amts. from the I with a free 4-position), which oxidized yielded the corresponding 4,

4'-dihydroxydiphenyl sulfones; the distillate

dissolved in a little MeOH, the solution treated with 5% aqueous HgCl2, the precipitate

(XXXVII) filtered off, and the filtrate worked up gave the corresponding reduced phenol; the XXXVII treated with Et2O-concentrated HCl and the Et2O layer

distilled gave the mercaptans, b0.3 60-100°. Thus was obtained XX, m. 38-40° (petr. ether); Me ether (XXXVIII) m. 45-6°. The I dissolved in the smallest amount of solvent (CH2Cl2 in this case), the solution added dropwise with shaking to liquefied H2S (10 ml./g. I) followed by Et3N (1 drop/g. I), the mixture allowed to stand a specified time (2 days in this case) with continuous cooling with Dry Ice, the H2Sevapd., and the residue distilled to 150°/0.3 mm. gave 16% o-cresol (XXXIX) and (by oxidation of the mixture) 36% XIX. The above experiment repeated, allowed to stand

10 days, the mixture reduced in the cold with Zn and acid, and worked up gave 46% XXXIX, 2% XVII, 3% 2,5-Me(HS)C6H3OH (XL), 10% XVIII, and 2% XIX. The reaction repeated with absolute MeOH, the mixture allowed to stand 8 days, reduced in the cold with Zn and acid, and worked up gave 54% XXXIX, 0.4% XVII, 6% XL, 5% XVIII, and 4% XIX. III treated 2.5 days with H2S in CH2Cl2, the H2S evaporated, the residue treated with EtOH, the precipitate (40%)

filtered off, washed with EtOH, and recrystd. twice from EtOH gave XLI, m. 165-70° (decomposition); from the filtrate was isolated 38% 2,4-Me2C6H3OH. MeSO2Cl (3-4 g. for each g. I to be used) reduced by the procedure for the preparation of EtSO2Na (Houben-Weyl-Muller, Methods Organic Chemistry, Stuttgart, 1955, IX, p. 292), the resulting sirupy solution of MeSO2Na acidified with 5% MeOH-HCl under ice cooling until weakly acid to Congo red, the precipitate filtered off, washed with MeOH, the filtrate and

CA

washings added to the I dissolved in the least amount of MeOH, the mixture allowed to stand 2 days, warmed 30 min., the MeOH distilled, the residue taken up in H2O, the solution scratched, allowed to stand some time in the cold, and the precipitate filtered off gave the product. Thus were obtained XXIII, m. 116-17° (dilute aqueous AcOH), and XXVII, m. 131-2° (aqueous MeOH). CH:CH.CMe:CH.C(OArc)2.CO (XLII) (2 g.) treated with MeSH solution (MeSH-MeOH-MeONa) as above, the mixture worked up, the oily product methylated in the cold, the mixture heated 15 min. on a H2O bath with excess aqueous NaOH, treated again with Me2SO4, worked up, the product distilled (b10 150-70°), and recrystd. from petr. ether gave 44 % 4,1,2,5-Me (MeO) 2 (MeS) C6H2, m. 58-9°, oxidized to 90% corresponding sulfone (XLIII), m. 140-1° (dilute AcOH). XLII (1.5 g.) treated with MeSO4H like the I and the product saponified and methylated gave 1.12 g. XLIII. The I treated with PhSH (2 moles/mole I) as a 20% solution of PhSNa in absolute MeOH, the product dissolved in aqueous alkali, filtered, the filtrate acidified, extracted with Et2O, the extract

evaporated, the residue distilled to 150°/10 mm. (reduction product and excess PhSH), and the residual viscous oil oxidized gave the sulfones. The I treated with PhSH using Et3N (CA 51, 12848a) and the products oxidized gave the sulfones. Thus were obtained XXXIII, m. 242-4.5° (EtOH), and XXXIV, m. 157-60° [Me ether m. 65-6.5° (dilute AcOH)]. IV treated with PhCH2SH with MeOH-MeONa (usual procedure) gave 8% mesitol and 45% 2,4,6,3-Me3(PhCH2S)C6HOH (crude), b0.3 160-200°, m. 70-1° (petr. ether), oxidized to the sulfone, m. 158-9°. II and XVIII treated with Et3N [by Kotlan and Wessely's procedure (loc. cit.) for the reaction of the I with PhSH with Et3N], the solvent evaporated, and the residue oxidized gave 60% XIX, m. 274-6° (AcOH). VI treated similarly with XXI gave 40% XXII, m. 303-6° (AcOH). The appropriate I treated with PhSO2H (by the procedure of K. and W., loc. cit.) gave XXXII, m. 128-9° (dilute AcOH), XXXIV, m. 86-7° (dilute AcOH), and XXXV, m. 156-8° (dilute AcOH). Comparison syntheses: O-Carbethoxyphenolsulfonyl chlorides were prepared from the corresponding phenolsulfonic acid di-Na salts [procedure of Karrer and Laiser (CA 39, 5197) for an analogous compound]. Sulfonyl chlorides of phenol ethers were prepared by treating the latter with ClSO3H (Kolhatkar and Bokil, CA 25, 2126). The sulfonyl chlorides were reduced to mercaptans by the procedure of Karrer and L. (CA 39, 5197). Oxidation of 2,4-Me(MeS)C6H3OMe (Shah, et al., CA 28, 1248) gave 76% sulfone, m. 71-2° (dilute AcOH). Oxidation of 2,5-Me(MeS)C6H3OMe (loc. cit.) gave 78% sulfone, m. 104-5° (dilute AcOH). From 2,4-Me(NaO3S)C6H3ONa (Hultquist, et al., CA 46, 6608h) was prepared 69% 2,4-Me(NaO3S)C6H3OCO2Et (XLIV). From crude XLIV was prepared 91% 2,4-Me(ClO2S)C6H3OCO2Et (XLV), m. 48-9° (petr. ether). Crude XLV reduced, saponified, and the product (b10 130-60°) recrystd. several times from petr. ether gave XVIII, m. 41-2°. 3,5,2-Me2(MeO)C6H2NH2 (CA 51, 12848a) diazotized in H2SO4 solution, the diazonium solution converted by SO2 (procedure of Shah, et al.,

28, 1248) to crude 2,4,6-Me2(HO2S)C6H2OMe, the latter reduced, the mixture steam distilled, and the product distilled in vacuo gave 44% 2,4,6-Me2(HS)C6H2OMe (XLVI), b10 100-2°. XLVI methylated, the product (91%) distilled (b10 120-50°), and recrystd. from petr. ether gave XXXVIII, m. 45-6°, oxidized to 77% sulfone, m. 55-6° (dilute AcOH). 2,4,5-Me2(O2N)C6H2SO3Na hydrogenated with Raney Ni at 60°/50 atmospheric H pressure, filtered, the filtrate treated with 40 g. H2SO4, the solution treated with N oxide gas with ice-cooling and stirring until the solution colored KI-starch paper blue, added to hot dilute H2SO4, after N evolution ceased the mixture neutralized with BaCO3, filtered, the filtrate concentrated to 200 ml., treated with 13 g. NaOH and 35

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q. ClCO2Et, the solution evaporated to dryness, the residue ground with 100 q.
    PCl5, the mixture decomposed with ice H2O, and the precipitate filtered off
    q. 2,4,5-Me2(ClO2S)C6H2OCO2Et (XLVII), amorphous; a portion extracted with
    boiling petr. ether and recrystd. using C gave XLVII, m. 52.5-4.0°.
         Crude XLVII reduced, saponified, and the product distilled
    gave 4.8 g. 2,4,5-Me2(HS)C5H2OH, b10 120-50°, m. 90-2°,
    methylated to XXXVI, m. 50-1°. 2,4-Me2C6H3OMe converted with
    C1SO3H to the 5-SO2Cl derivative, the latter reduced, the product
     isolated by steam distillation, purified by distillation (b10 120-50°), and
    recrystd. from petr. ether gave 2,4,5-Me2(HS)C6H2OMe, m. 37-9°
     (petr. ether), methylated to XXXVI. From 2,5-Me2C6H3OH was prepared 99%
    2,5,4-Me2(NaO3S)C6H2ONa (XLVIII) (Hultquist, et al., CA 46, 6608h).
    crude XLVIII was prepared 58% 2,5,4-Me2(NaO3S)C6H2OCO2Et, converted
     to 40% 2,5,4-Me2)ClO2S)C6H2OCO2Et (XLIX), m. 77-8° (petr. ether).
    XLIX reduced, saponified, and the product distilled (130-60°/10
    mm.) gave 75% 2,5,4-Me2(HS)C6H2OH (L), m. 93-4° (petr. ether).
     and equimolar amts. of MeI and NaOEt in EtOH heated 3 hrs. at 60°
     in a bomb tube gave 44% 2,5,4-Me2(MeS)C6H2OH, m. 96-7° (petr.
     ether), oxidized to 52% XXV, m. 143-4° (dilute AcOH). L methylated
     with MeI as above gave 62% XVI, m. 59-61° (petr. ether), oxidized
     to 62% sulfone, m. 156-7° (dilute AcOH). 4,1,2-Me(MeO)2C6H3 treated
     with ClSO3H gave 94% 5-SO2Cl derivative, m. 78-80°, reduced to
     4,1,2,5-Me(MeO)2(HS)C6H2, m. 58-9° (petr. ether), methylated to 89%
     4,1,2,5-Me (MeO) 2 (MeS) C6H2. 2,6-Et2C6H3OH oxidized with Pb(OAc) 4
     (Metlesics, et al., CA 52, 11775a) gave 52% VII, b0.2 82-4°. VII
     treated with PhMgBr (method of Wessely, et al., CA 47, 9936a), the mixture
     steam distilled in vacuo, and the product distilled gave 3.4 g.
     2,6,3-Et2PhC6H2OH (LI), b0.1 125-35°. LI (3.5 g.) oxidized with
     Pb(OAc)4 in CHCl3 gave 2.14 g. VIII, m. 105-6°, which gave by
     Thiele rearrangement (cf. Wessely and Metlesics, CA 49, 9529c) 73%
     2,6,5,1,3-Et2Ph(HO)2C6H, m. 112-13°, not oxidized by FeCl3 to a
     quinone.
=> d his all
     (FILE 'HOME' ENTERED AT 12:12:37 ON 26 JUL 2005)
     FILE 'REGISTRY' ENTERED AT 12:12:50 ON 26 JUL 2005
                E DIHYDROXYDIPHENYLSULFONE/CN 5
                E TRIHYDROXYTRIPHENYLSULFONE/CN 5
             47 S DIHYDROXY (L) DIPHENYLSULFONE
L1
              0 S TRIHYDROXY (L) TRIPHENYLSULFONE
L2
L3
              0 S TRIHYDROXY (L) TRIPHENYLSULPHONE
              0 S TRIHYDROXY(L)?PHENYLSULFONE
L4
     FILE 'MEDLINE, BIOSIS, EMBASE, CAPLUS' ENTERED AT 12:15:10 ON 26 JUL 2005
              3 FILE MEDLINE
L5
L6
              2 FILE BIOSIS
              1 FILE EMBASE
L7
           1120 FILE CAPLUS
L8
     TOTAL FOR ALL FILES
Ь9
           1126 S (L1 OR DIHYDROXYDIPHENYLSULFONE OR DIHYDROXYDIPHENYLSULPHONE)
L10
              O FILE MEDLINE
              0 FILE BIOSIS
L11
L12
              O FILE EMBASE
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O S TRIHYDROXYTRIPHENYLSULFONE OR TRIHYDROXY(L)TRIPHENYLSULFONE O

0 FILE CAPLUS

TOTAL FOR ALL FILES

L13

L14

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Page 20
            O FILE MEDLINE
L15
L16
            0 FILE BIOSIS
            O FILE EMBASE
L17
L18
            1 FILE CAPLUS
    TOTAL FOR ALL FILES
      1 S ?TRIPHENYLSULFONE? OR ?TRIPHENYLSULPHONE?
L19
L20
            O FILE MEDLINE
             0 FILE BIOSIS
L21
            O FILE EMBASE
L22
            O FILE CAPLUS
L23
    TOTAL FOR ALL FILES
L24
     0 S L9 AND L19
L25
            O FILE MEDLINE
L26
            0 FILE BIOSIS
L27
            O FILE EMBASE
L28
           46 FILE CAPLUS
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L29
      46 S (DISSOLV? OR SUSPEND?) AND L9
L30
            O FILE MEDLINE
L31
            0 FILE BIOSIS
L32
            0 FILE EMBASE
L33
            0 FILE CAPLUS
    TOTAL FOR ALL FILES
L34
           0 S ALKALI METAL HYDROXIDE AND L29
L35
             0 FILE MEDLINE
            0 FILE BIOSIS
L36
            0 FILE EMBASE
L37
L38
            4 FILE CAPLUS
    TOTAL FOR ALL FILES
      4 S CRUDE AND L29
L39
L40
             3 FILE MEDLINE
            1 FILE BIOSIS
L41
            2 FILE EMBASE
L42
L43
            1 FILE CAPLUS
    TOTAL FOR ALL FILES
            7 S WAKAYAMA F?/AU
L45
            32 FILE MEDLINE
L46
           26 FILE BIOSIS
L47
           29 FILE EMBASE
         188 FILE CAPLUS
L48
    TOTAL FOR ALL FILES
       275 S YANASE N?/AU
L50
          369 FILE MEDLINE
L51
          458 FILE BIOSIS
L52
          326 FILE EMBASE
L53
          994 FILE CAPLUS
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L54
     2147 S KITAHARA T?/AU
L55
            O FILE MEDLINE
L56
             5 FILE BIOSIS
L57
            O FILE EMBASE
L58
          21 FILE CAPLUS
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L59
     26 S NATE N?/AU
L60
            7 FILE MEDLINE
L61
            10 FILE BIOSIS
L62
            2 FILE EMBASE
L63
            28 FILE CAPLUS
     TOTAL FOR ALL FILES
            47 S OI F?/AU
L64
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Page 21
L65
              O FILE MEDLINE
L66
              0 FILE BIOSIS
L67
              O FILE EMBASE
L68
              2 FILE CAPLUS
     TOTAL FOR ALL FILES
L69
              2 S L64 AND L59 AND L54 AND L49
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                E "2,4'-DIHYDROXYDIPHENYL SULFONE"/CN
L70
              4 S E3-E6
                E "4,4'-DIHYDROXYDIPHENYL SULFONE"/CN 5
L71
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L73
             32 FILE BIOSIS
L74
              0 FILE EMBASE
L75
           1750 FILE CAPLUS
     TOTAL FOR ALL FILES
L76
           1782 S L70 OR L71
L77
              1 FILE MEDLINE
L78
              1 FILE BIOSIS
L79
              3 FILE EMBASE
L80
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L81
            755 S "4,4'-DIHYDROXYDIPHENYL SULFONE"
L82
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L83
              0 FILE BIOSIS
L84
              0 FILE EMBASE
L85
            170 FILE CAPLUS
     TOTAL FOR ALL FILES
L86
            170 S "2,4'-DIHYDROXYDIPHENYL SULFONE"
L87
              O FILE MEDLINE
L88
              O FILE MEDLINE
L89
              8 FILE BIOSIS
L90
              1 FILE EMBASE
L91
            737 FILE CAPLUS
     TOTAL FOR ALL FILES
L92
            746 S (L76 OR L81 OR L86) AND (MAKE OR MAKING OR PROCESS? OR PRODUC
L93
              O FILE MEDLINE
L94
              0 FILE BIOSIS
L95
              0 FILE EMBASE
L96
              0 FILE CAPLUS
     TOTAL FOR ALL FILES
              0 S TRI HYDROXY TRIPHENYL SULFONE OR TRIHYDROXY TRIPHENYL SULFONE
L97
L98
              O FILE MEDLINE
L99
              0 FILE BIOSIS
L100
              1 FILE EMBASE
L101
              6 FILE CAPLUS
     TOTAL FOR ALL FILES
L102
              7 S TRIPHENYLSULFONE OR TRIPHENYL SULFONE OR TRIHYDROXY(L) (SULFON
L103
              O FILE MEDLINE
L104
              0 FILE BIOSIS
L105
              O FILE EMBASE
L106
              0 FILE CAPLUS
     TOTAL FOR ALL FILES
L107
              0 S L102 AND L92
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Page 22

L108	8 0 FILE MEDLINE			
L109	9 0 FILE BIOSIS			
L110	0 0 FILE EMBASE			
L111	1 8 FILE CAPLUS	•		
	TOTAL FOR ALL FILES			
L112	2 8 S L92 AND CRYST	ral?	AND	CRUDE
L113	3 O FILE MEDLINE			
L114	4 0 FILE BIOSIS			
L115	5 0 FILE EMBASE			
L116	6 5 FILE CAPLUS			
	TOTAL FOR ALL FILES			
L117	7 5 S L112 NOT (L39	OR	L69)	•

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL
FULL ESTIMATED COST	74.45	SESSION 202.41
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE ENTRY	TOTAL SESSION
CA SUBSCRIBER PRICE	-3.65	-8.03

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